(11) EP 0 517 104 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention of the grant of the patent:27.11.1996 Bulletin 1996/48

(51) Int Cl.⁶: **C07D 251/70**, C08K 5/3492, A61K 7/42

- (21) Application number: 92109017.1
- (22) Date of filing: 29.05.1992
- (54) 1,3,5-Triazine derivatives, their preparation and use as sunscreens 1,3,5-Triazinderivate, ihre Herstellung und ihre Verwendung als Lichtschutzmittel Dérivés de 1,3,5-triazines, leur préparation et leur utilisation comme filtres solaires
- (84) Designated Contracting States:

 AT BE CH DE DK ES FR GB GR IT LI LU MC NL
 PT SE
- (30) Priority: 04.06.1991 IT MI911519
- (43) Date of publication of application: 09.12.1992 Bulletin 1992/50
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SEIFEN,OLE,FETTE,WACHSE vol. 115, no. 18,21
 November 1989, AUGSBURG, DEUTSCHLAND, pages 661-662; DR.K.SPERLING: "UV-Filter für Haut- und Produktschutz in kosmetischen Formulierungen."

Remarks:

The file contains technical information submitted after the application was filed and not included in this specification

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Description

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The present invention relates to 1,3,5-triazine derivatives of the general formula:

10 (I) 15

in which

20 R is C₅-C₁₂cycloalkyl which may be mono- or polysubstituted by C₁-C₄alkyl, or a group of the formula (II), (III) or (IV):

25 30 (II) (III) (IV)

in which R2 is C1-C9alkyl,

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n can be an integer from 0 to 3, R₃ is hydrogen or methyl,

A is C5-C8cycloalkyl or C4-C8alkyl,

B is C1-C4alkyl,

m can be an integer from 1 to 10, and

R' and R" which are the same or different, can have the same meaning as R or they are hydrogen, an alkali metal, an ammonium group which may be substituted by alkyl or hydroxyalkyl radicals, or C1-C18alkyl,

and to a process for their preparation and to their use as light stabilisers.

As is known, the ultraviolet radiation in sunlight exerts a damaging action on skin tissue and causes degradation of polymers. By means of using particular compounds, so-called sunscreens, which are capable of absorbing the UV part of solar radiation, it is possible to inhibit or at least to slow down its noxious effects and the ageing of skin and of synthetic polymer materials.

Numerous substances have been studied as protective agents and used in experiments, and there is extensive patent literature on this subject, wherein compounds belonging to various chemical classes are proposed, which are capable of absorbing in the ultraviolet region and particularly the most noxious radiation between 290 and 320 nm, socalled here UVB.

Of these compounds, however, only relatively few have proved suitable for application in practice, including esters of p-methoxycinnamic acid and p-dimethylaminobenzoic acid, benzotriazoles and hydroxybenzophenones.

In SOFW, 115(1989) 21, November no.18, the compound octyl triazone (Uvinul T150) useful as UVB absorber is described.

A common disadvantage of all these compounds is their low absorbance, so that it is necessary to use relatively

large quantities to obtain the best light-protective capacity, that is to say their so-called sun "protection factor" is relatively low. The sun protection factor (SPF), is a unit of measurement of the light-protective power of a sunscreen or of a cosmetic formulation containing one or more sunscreens. It is directly correlated to the specific extinction and is determined in vivo, by tests on human subjects.

These tests are very time-consuming and expensive, so that various attempts have been made to develop techniques which are suitable for determining the SPF in vitro. One method of determining the SPF in vitro, which has given sufficiently reliable results, is that described by B.L. Diffey and J. Robson in J. Soc. Cosmet. Chem. 40, 127-133 (1989). This method consists in using a synthetic material, Transpore, as a substitute for human skin and in spectro-radiometric measurement of the UV radiation from a sunscreen, applied to the above mentioned substrate. The results thus obtained with sunscreens which are known and already used in cosmetics, are very close to those obtained experimentally in vivo.

In the Patent DE 3,206,398, novel sunscreens are described which are derived from s-triazine and are obtained by reacting dichlorotriazine with esters of p-aminobenzoic acid. These compounds absorb very intensively in the UVB zone and consequently show a much higher SPF than previously known sunscreens.

It has now been found that the compounds of the invention have an unexpectedly high SPF, higher than that of the products of DE 3,206,398. A further subject of the invention is therefore the use of the compounds of the formula (I) as sunscreens and light stabilisers.

The compounds of the invention can be usefully employed in the light stabilisation of synthetic polymers, against the solar radiation band between 290 and 320 nm, in order to avoid photodegradation and alterations. It has also been found that the compounds of the formula (I) have a surprising high protective activity on skin against the damaging component of solar radiation.

The compounds according to the present invention can be prepared by reacting symmetrical trichlorotriazine or tribromotriazine with esters of p-aminobenzoic acid of the formula (V)

in which R₄ is as defined above for R, R' and R".

Suitable solvents, in which the reaction can be carried out, are those which are inert towards the reagents, for example acetonitrile, ketones such as acetone and methyl ethyl ketone, ethers such as diethyl ether, diisopropyl ether, tetrahydrofuran and dioxane, and aliphatic or aromatic hydrocarbons such as pentane, heptane, cyclohexane, benzene, toluene, xylene or mixtures thereof.

The reaction can be carried out in the absence or presence of acid acceptors, for example hydroxides of alkali metals or alkaline earth metals, or bicarbonates or carbonates of alkali metals, in molar ratios of 3-4 mole of ester of p-aminobenzoic acid per one mole of trichlorotriazine.

If the substituents R, R' and R" differ from one another, the reaction between the trihalogenotriazine and the different esters is carried out in stages, if appropriate with isolation, and with purification, if necessary, of the intermediate stage before the subsequent reaction with further ester of p-aminobenzoic acid; however, these synthesis methods and reactions of trihalogenotriazines for the substitution of three halogen atoms by identical or different amine radicals are well known and widely described in the technical literature, especially in the literature dealing with certain types of dyestuffs and optical brighteners.

Some of the esters of the formula (V) are known, and others which are novel are prepared by known methods.

The compounds according to the present invention possess, in addition to very high absorption in the UVB zone, other characteristics which are regarded as necessary for application in practice, for example light stability, heat stability, non-toxicity and so on. In fact, an optimum UVB absorber should have the following properties:

- 1) High specific extinction E' at 290-320 nm, which means low dosages in use and therefore cost-effectiveness and minimum toxicological risk.
- 2) Light stability.

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- 3) Heat stability.
- 4) Oxidation stability.
- 5) Stability at varying pH.
- 6) Good solubility, emulsifiability or dispersibility in base substances commonly used as substrates for dermato-

logical formulations.

- 7) Negligible toxicity.
- 8) No significant characteristic colour or odour.
- 9) Relatively high molecular weight, which means a lower probability of absorption by the skin and therefore greater safety from the toxicological viewpoint.
- 10) Compatibility with the various substances generally used in dermatological formulations.

According to one of the preferred embodiments of the invention, the compositions containing the compounds of the formula (I) are used for protecting the skin from the damaging effects of solar radiations.

The compounds according to the present invention can be added, of course also in combination with other stabilisers, to cosmetic formulations and also to synthetic polymers generally in quantities of between 0.05 and 10%, preferably from 0.1 to 5%, by weight of the polymer or cosmetic preparation.

The cosmetic preparations can be of various types and can be used for various purposes. Generally they take the form of ointments, creams, lotions and emulsions.

The compounds of the formula I are added either for protecting the preparations themselves, for example to avoid undesired discolourations, or for protecting the epidermis treated with the cosmetic preparation against the damaging action of UVB radiation, which causes erythema and accelerates ageing of the skin, so that it becomes prematurely dry, wrinkled or scaly.

The examples which follow illustrate the present invention.

EXAMPLE 1

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4.6 g of cyanuric chloride and 17.5 g of cyclohexyl p-aminobenzoate are added to 150 ml of xylene. The reaction mixture is stirred under reflux for 6 hours. 100 ml of octane are added, and the mixture is cooled to 0-5°C. The white crystallised product is filtered off, washed and dried in vacuo.

This gives 16 g of 2,4,6-tris-[p-(cyclohexyloxycarbonyl)-anilino]-s-triazine, melting point: 171-173°C UV (methanol): E' - 1691 at 312 nm

EXAMPLES 2-7

The compounds listed in Table 1 are prepared according to the procedure described in Example 1.

TABLE 1

10	ROOC-	-NHN	NET-		
20	Example	R = R' = R*	Melting point	E;	nm
25	2	CH ₃ CH ₃	268-270	1493	312
30	3	-CH3	152-155	1591	312
35	4	CH3-CH-CH3	122-125	1412	313
40	5	-C(CH ³) ³	> 260	1386	312
45	6 -0	H2-CH2-O-(O)	155-158	1465	313
50	7 –(CH-CH ₂ -O-CH ₃	150-153	1763	312

EXAMPLE 8

6.7 g of sodium bicarbonate are added to a solution of 13.8 g of cyanuric chloride in 150 ml of acetone, cooled to 0°C. A solution of 27.7 g of di-[2-(4-tert-octylphenoxy)-ethyl] 4-aminobenzoate in 100 ml of acetone is slowly added to the mixture obtained, maintaining the temperature at 0-2°C.

The mixture is stirred for 30 minutes at 0°C, and 50 ml of water are then slowly added. This gives a very dense suspension which is stirred for a further 30 minutes at 0°C and then filtered, and the filtercake is washed many times with water and then with acetone and dried at 40°C in vacuo. This gives 36 g of 2-[p-[2-(4-tert-octylphenoxy)-ethoxy-carbonyl]-anilino]-4,6-dichloro-s-triazine. Melting point: 249-251°C.

12.8 g of methyl p-aminobenzoate are added to 20.7 g of this intermediate in 200 ml of xylene, and the mixture is stirred for 5 hours under reflux. This xylene is then distilled off, and the residue is crystallised from isopropanol. This gives 19 g of 2-[p-[2-(4-tert-octylphenoxy)-ethoxycarbonyl]-anilino]-4,6-bis-[p-(methoxycarbonyl)-anilino]-s-triazine. Melting point: 157-160°C

UV (methanol):

E' = 1636 at 312.6 nm.

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EXAMPLES 9-17

Using a similar procedure as described in Example 8, the compounds of Table 2 are prepared.

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TABLE 2

10		ROOC-NH -	N = NH		OR'	
15	Example	R	R' = Ru	Melting Point	. E'	n m
20	9	сн ³ -сн-сн ³	CH3	217-220	1954	312
30	10	-cH2-CH3-0 C(CH3)3	снз	127-130	1675	313
35	11	-cH2-CH3-0-C(CH3)3	снз	193-195	1761	313
40	12	——	-CH2-CH-C4H9	85-88	1500	313
45	13	→	-CH2-CH-C4H9 C2H5	144-147	1615	313
50 55	14	CH3 CH3	-сн ₃	181-184	170 9	313

TABLE 2 (cont.)

Example	R	R' = R*	Melting Point	E,	nea
15	−CH3	-сн ₃	153-156	1807	312
16	CH3-CH-CH3	c ₄ н ₉	161-163	1748	313
17	——— сн ³	c ₄ n ₉	166-168	1719	313

EXAMPLE 18

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The SPF of some compounds from the examples described are determined using the method described by B.L. Diffey and J. Robson in J. Soc. Cosmet. Chem. 40, 127-133 (1989).

The results obtained are listed in the table which follows.

TABLE 3

Example	SPF
Compound from Example 3	7.6
Compound from Example 4	8.2
Compound from Example 10	8.5
Compound from Example 13	7.7
Compound from Example 14	8.5
Compound from Example 15	8.4
Compound from Example 16	8.3
Compound A	7.4

Compound A is Example 1 described in Patent DE 3,206,398.

EXAMPLE 19

50 Preparation of a sun cream

A mixture composed of 10 g of cyclodimeticone/dimeticone copolymer (Dow Corning Q2-3225), 10 g of cyclometicone (Dow Corning 344), 0.5 g of polysorbate 20 (Tween 20) and 3 g of the compound of Example 3 is prepared. This mixture is added to a previously prepared solution composed of 0.2 g of 1,1'-methylene-bis3-(3'-hydroxymethyl-2,4-dioxo-imidazolidinyl)-urea, 0.05 g of methylparaben (methyl p-hydroxybenzoate) and 73.25 g of water.

Claims

1. Compounds of the general formula (I):

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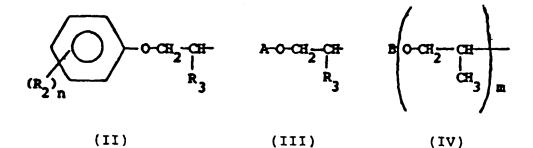
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20 in which

R is C_5 - C_{12} cycloalkyl which may be mono- or polysubstituted by C_1 - C_4 alkyl, or a group of the formula (II), (III) or (IV):

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in which R₂ is C₁-C₉alkyl,

n can be an integer from 0 to 3,

R₃ is hydrogen or methyl,

A is C5-C8cycloalkyl or C4-C8alkyl,

B is C₁-C₄alkyl,

m can be an integer from 1 to 10, and

R' and R" which are the same or different, can have the same meaning as R or they are hydrogen, an alkali metal, an ammonium group which may be substituted by alkyl or hydroxyalkyl radicals, or C₁-C₁₈alkyl.

2. A compound according to Claim 1, selected from the group comprising:

2,4,6-tris-[p-(cyclohexyloxycarbonyl)-anilino]-s-triazine;

2,4,6-tris-[p-(3,3,5-trimethylcyclohexy)-anilino]-s-triazine;

2,4,6-tris-[p-(methylcyclohexyloxycarbonyl)-anilino]s-triazine;

2,4,6-tris-[p-(2-isopropyl-5-methylcyclohexyloxy-carbonyl)-anilino]-s-triazine;

2,4,6-tris-[p-(4-tert-butylcyclohexyloxycarbonyl)-anilino]-s-triazine

2,4,6-tris-[p-(2-phenoxyethoxycarbonyl)-anilino]-s-triazine;

2,4,6-tris-[p-(2-methoxy-1-methylethoxycarbonyl)-anilino]-s-triazine;

2-[p-[2-(4-tert-octylphenoxy)-ethoxycarbonyl]-anilino]-4,6-bis-[p-(methoxycarbonyl)-anilino]-s-triazine;

2-[p-[2-isopropyl-5-methylcyclohexyloxycarbonyl]-anilino]-4,6-bis-[p-(methoxycarbonyl)-anilino]-s-triazine;

 $\hbox{$2$-[p-[2-(2,4-ditert-butylphenoxy)-ethoxycarbonyl]-anilino]-4,6-bis-[p-(methoxycarbonyl)-anilino]-s-triazine;}$

2-[p-(2-ethylhexyloxycarbonyl]-anilino]-4,6-bis-[p-(methylcyclohexyloxycarbonyl)-anilino]-s-triazine:

2-[p-(2-ethylhexyloxycarbonyl)-anilino]-4,6-bis-[p-(methylcyclohexyloxycarbonyl)-anilino]-s-triazine;

2-[p-(methoxycarbonyl)-anilino]-4,6-bis-[p-(3,3,5-trimethylcyclohexyloxycarbonyl)-anilino]-s-triazine;

2-[p-(methoxycarbonyl)-anilino]-4,6-bis-[p-(methylcyclohexyloxycarbonyl)-anilino]-s-triazine;

 $\hbox{$2$-[p-(2-isopropyl-5-methylcyclohexyloxycarbonyl)-anilino]-4,6-bis-[p-(butoxycarbonyl)-anilino]-s-triazine, and in the property of the pr$

2-[p-(butoxycarbonyl)-anilino]-[p-(methylcyclohexyloxycarbonyl)-anilino]-s-triazine.

3. Process for preparing the compounds according to Claim 1, characterised in that a compound of the formula (VI):

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where X is chlorine or bromine, is reacted with an ester of 4-aminobenzoic acid of the formula (V):

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(.VI)

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where R₄ is as defined above for R, R' and R".

4. Use of the compounds according to Claim 1 as stabilisers for synthetic polymers.

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5. Use of the compounds according to Claim 1 as agents useful in dermatology.

6. Use of the compounds according to Claim 1 as sunscreens and free-radical deactivators for the preparation of cosmetic and dermatological compositions which can be used for protection of the skin.

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7. Cosmetic and dermatological compositions containing a compound according to claims 1, 2 as the active component.

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8. Cosmetic and dermatological compositions according to Claim 7, containing 0.05 to 10% by weight of active principle.

9. Cosmetic and dermatological compositions according to Claim 7, containing 0.1 to 5% by weight of active principle.

45 Patentansprüche

1. Verbindungen der allgemeinen Formel (I)

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R für C₅-C₁₂-Cycloalkyl steht, welches durch C₁-C₄-Alkylreste mono- oder polysubstituiert sein kann, oder eine Gruppe der Formel (II), (III) oder (IV):

worin R2 gleich C1-C9-Alkyl ist,

n eine ganze Zahl von 0 bis 3 bedeutet,

R₃ ein Wasserstoffatom oder einen Methylrest darstellt,

A für C5-C8-Cycloalkyl oder C4-C8-Alkyl steht,

B gleich C₁-C₄-Alkyl ist,

m eine ganze Zahl von 1 bis 10 bedeutet, und

R' und R', die gleich oder unterschiedlich sind, die selbe Bedeutung wie R haben oder Wasserstoff, ein Alkalimetall, einen Ammoniumrest, der gegebenenfalls durch Alkyl- oder Hydroxyalkyl-Reste substituiert ist, oder C_1 - C_{18} -Alkyl bedeuten.

2. Verbindungen nach Anspruch 1 aus der Gruppe von

2,4,6-Tris-[p-(cyclohexyloxycarbonyl)-anilino]-s-triazin;

2,4,6-Tris-[p-(3,3,5-trimethylcyclohexyloxycarbonyl)-anilino]-s-triazin;

2,4,6-Tris-[p-(methylcyclohexyloxycarbonyl)-anilino]-s-triazin;

2,4,6-Tris-[p-(2-isopropyl-5-methylcyclohexyloxy-carbonyl)-anilino]-s-triazin;

2,4,6-Tris-[p-(4-tert-butylcyclohexyloxycarbonyl)anilino]-s-triazin;

2,4,6-Tris-[p-(2-phenoxyethoxycarbonyl)-anilino]-s-triazin;

2,4,6-Tris-[p-(2-methoxy-1-methylethoxycarbonyl)-anilino]-s-triazin;

2-[p-[2-(4-Tert-octylphenoxy)-ethoxycarbonyl]-anilino]-4,6-bis-[p-(methoxycarbonyl)-anilino]-s-triazin;

2-[p-[2-Isopropyl-5-methylcyclohexyloxycarbonyl]-anilino]-4,6-bis-[p-(methoxycarbonyl)-anilino]-s-triazine;

2-[p-[2-(2,4-Ditert-butylphenoxy)-ethoxycarbonyl)-anilino]-4,6-bis-[p-(methoxycarbonyl)-anilino]-s-triazin;

2-[p-(2-Ethylhexylcarbonyl] -anilino]-4,6-bis-[p-(methylcyclohexyloxycarbonyl)-anilino]-s-triazine;

2-[p-(2-Ethylhexyloxycarbonyl)-anilino]-4,6-bis-[p-(methylcyclohexyloxycarbonyl)-anilino]-s-triazin;

2-[p-(Methoxycarbonyl)-anilino]-4,6-bis-[p-3,3,5-trimethylcyclohexyloxycarbonyl)-anilino]-s-triazin;

2-[p-(Methoxycarbonyl)-anilino]-4,6-bis- [p- (methylcyclohexyloxycarbonyl)-anilino]-s-triazin;

2-[p-(2-Isopropyl-5-methylcylohexyloxycarbonyl)-anilino]-4,6-bis-[p-(butoxycarbonyl)-anilino]-s-triazin;

2-[p-(Butoxycarbonyl)-anilino]-[p-(methylcyclohexyloxycarbonyl)-anilino]-s-triazin.

3. Verfahren zur Herstellung der Verbindungen nach Anspruch 1, dadurch gekennzeichnet, daß eine Verbindung der

allgemeinen Formel (VI)

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worin X für Chlor oder Brom steht, mit einem Ester der 4-Aminobenzoesäure der allgemeinen Formel (V)

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$$H_2N$$
—COOR₄

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worin R4 gemäß der obigen Definition für R, R' und R" ist, umgesetzt wird.

- Verwendung der Verbindungen nach Anspruch 1 als Stabilisatoren für synthetische Polymere.
 - 5. Verwendung der Verbindungen nach Anspruch 1 als für die Dermatologie geeignete Mittel.
- 6. Verwendung der Verbindungen nach Anspruch 1 als Sonnenschutz und Desaktivatoren für freie Radikale zur Her-30 stellung von kosmetischen und dermatologischen Zusammensetzungen, welche für den Schutz der Haut verwendet werden können.
 - 7. Kosmetische und dermatologische Zusammensetzungen, welche eine Verbindung nach den Ansprüchen 1 oder 2 als aktive Wirkstoffe enthalten.

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- 8. Kosmetische und dermatologische Zusammensetzungen nach Anspruch 7, welche 0,05 bis 10 Gew% des aktiven Wirkstoffes aufweisen.
- Kosmetische und dermatologische Zusammensetzungen nach Anspruch 7, welche 0,1 bis 5 Gew% des aktiven
 Wirkstoffs aufweisen.

Revendications

Composés de formule générale (i) :

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dans laquelle:

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R est un groupe cycloalkyle en C_{5-12} qui peut être mono- ou polysubstitué par un groupe alkyle en C_{1-4} , ou un groupe de formule (II), de formule (III) ou de formule (IV):

$$(R_2)_n \qquad (II) \qquad (III) \qquad (IV)$$

dans lesquelles:

R₂ est un groupe alkyle en C₁₋₉,

n peut être un entier de 0 à 3,

R₃ est un atome d'hydrogène ou un groupe méthyle,

A est un groupe cycloalkyle en C_{5-8} ou un groupe alkyle en C_{4-8} ,

B est un groupe alkyle en C₁₋₄,

m peut être un entier de 1 à 10, et

R' et R", qui sont identiques ou différents, peuvent avoir la même signification que R, ou ils sont un atome d'hydrogène, un métal alcalin, un groupe ammonium qui peut être substitué par un groupe alkyle ou hydroxyalkyle, ou un groupe alkyle en C₁₋₁₈.

2. Composé suivant la revendication 1, choisi dans le groupe comprenant :

2,4,6-tris-[p-(cyclohexyloxycarbonyl)-anilino]-s-triazine;

2,4,6-tris-[p-(3,3,5-triméthylcyclohexyloxycarbonyl)-anilino]-s-triazine;

2,4,6-tris-[p-(méthylcyclohexyloxycarbonyl)-anilino]-s-triazine;

2,4,6-tris-[p-(2-isopropyl-5-méthylcyclohexyloxycarbonyl)-anilino]-s-triazine;

2,4,6-tris-[p-(4-t-butylcyclohexyloxycarbonyl)-anilino]-s-triazine;

2,4,6-tris-[p-(2-phénoxyéthoxycarbonyl)-anilino]-s-triazine;

 $2,4,6-tris-[p-(2-m\'{e}thoxy-1-m\'{e}thyl\'{e}thoxycarbonyl)-anilino]-s-triazine;$

2-{p-[2-(4-t-octylphénoxy)-éthoxycarbonyl]-anilino}-4,6-bis-[p-(méthoxycarbonyl)-anilino]-s-triazine;

2-{p-[2-isopropyl-5-méthylcyclohexyloxycarbonyl]-anilino}-4,6-bis-[p-(méthoxycarbonyl)-anilino]-s-triazine;

 $2-\{p-[2-(2,4-di-t-buty|ph\acute{e}noxy)-\acute{e}thoxycarbonyl]-anilino\}-4,6-bis-[p-(m\acute{e}thoxycarbonyl)-anilino]-s-triazine;$

2-[p-(2-éthylhexyloxycarbonyl)-anilino]-4,6-bis-[p-(méthylcyclohexyloxycarbonyl)-anilino]-s-triazine;

2-[p-(méthoxycarbonyl)-anilino]-4,6-bis-[p-(3,3,5-triméthylcyclohexyloxycarbonyl)-anilino]-s-triazine;

2-[p-(méthoxycarbonyl)-anilino]-4,6-bis-[p-(méthylcyclohexyloxycarbonyl)-anilino]-s-triazine;

2-[p-(2-isopropyl-5-méthylcyclohexyloxycarbonyl)-anilino]-4,6-bis-[p-(butoxycarbonyl)-anilino]-s-triazine; 2-[p-(butoxycarbonyl)-anilino]-4,6-bis-[p-(méthylcyclohexyloxycarbonyl)-anilino]-s-triazine.

3. Procédé pour la préparation des composés suivant la revendication 1, caractérisé en ce qu'un composé de formule (VI):

dans laquelle X est un atome de chlore ou de brome, est traité avec un ester d'acide 4-aminobenzoïque de formule

dans laquelle R₄ est tel que défini plus haut pour R, R' et R".

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(V):

- 4. Utilisation des composés suivant la revendication 1 en tant que stabilisants pour des polymères synthétiques.
- 5. Utilisation des composés suivant la revendication 1 en tant qu'agents utiles en dermatologie.
- Utilisation des composés suivant la revendication 1 en tant que filtres solaires et désactivateurs de radicaux libres pour la préparation de compositions cosmétiques et dermatologiques qui peuvent être utilisées pour la protection de la peau.
 - 7. Compositions cosmétiques et dermatologiques contenant un composé suivant les revendications 1 et 2 en tant que composant actif.
- 40 8. Compositions cosmétiques et dermatologiques suivant la revendication 7, contenant 0,05 à 10% en poids de composant actif.
- Compositions cosmétiques et dermatologiques suivant la revendication 7, contenant 0,1 à 5% en poids de composant actif.